

533 Rec'd PCT/PTO

09 AUG 2000

ATTORNEY'S DOCKET NUMBER:

6-1034-040

**TRANSMITTAL LETTER TO THE UNITED STATES
DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 35 U.S.C. 371**

U.S. APPLICATION NO. (If known, see 37 CFR 1.5) (Not Yet Assigned - U.S. National Phase of Int'l PCT No. PCT/IB99/00207 filed February 8, 1999 claiming priority of EP 98420024.6 filed February 9, 1998)

INTERNATIONAL APPLICATION
NO. PCT/IB99/00207

INTERNATIONAL FILING DATE
February 8, 1999

PRIORITY DATE CLAIMED
February 9, 1998

TITLE OF INVENTION: MASKING AGENT IN POWDER FORM FOR PHARMACEUTICAL FLAVOURS

APPLICANT(S) FOR DO/EO/US Vasilios KANELLOPOULOS, Isabelle LOUIS-JOSEPH-DOGUE, Vincent Daniel McGINNISS, Durvodham MANGARAJ and Tomoki Tsuchiya NAKAMURA

Applicant herewith submits to the United States Designated/Elected Office(DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(l).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
 - a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☒ has been transmitted by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☐ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☐ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
 - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☐ have been transmitted by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☐ have not been made and will not be made
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)) (Unexecuted).
10. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 16. below concern other document(s) or information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.56, 1.97 and 1.98 with PTO Form 1449 attached; without Cited References:
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A **FIRST** preliminary amendment.
☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
14. ☐ A substitute specification.
15. ☐ A change of power of attorney and/or address letter.

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16. ☒ Other items or information:

PCT International Application Published Under the Patent Cooperation Treaty (PCT)

PCT Notification of the Recording of a Change Form PCT/IB/306

PCT Request

PCT Notification of the International Application Number and of the International Filing Date

PCT Written Opinion Form PCT/IPEA/408

PCT Notification of Transmittal of the International Preliminary Examination Report (PCT Fule 71.1) Form
PCT/IPEA/416

International Search Report

001021 21610360

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17. ☒ The following fees are submitted:

BASIC NATIONAL FEE (37 CFR 1.492(a)(1)-(5)):

Search report has been prepared by the EPO or JPO \$ 840.00

International preliminary examination fee paid to USPTO (37 CFR 1.482) \$

No international preliminary examination fee paid to USPTO (37 CFR 1.482
but international search fee paid to USPTO (37 CFR 1.445(a)(2)) \$Neither international preliminary examination fee (37 CFR 1.482) nor
international search fee (37 CFR 1.445(a)(2)) paid to USPTO \$International preliminary examination fee paid to USPTO (37 CFR 1.482)
and all claims satisfied provision of PCT Article 33(2)-(4) \$**ENTER APPROPRIATE BASIC FEE AMOUNT =** \$840.00Surcharge of \$130.00 for furnishing the oath or declaration later than
☐ 20 ☒ 30 months from the earliest claimed priority date (37 CFR 1.492(e)).

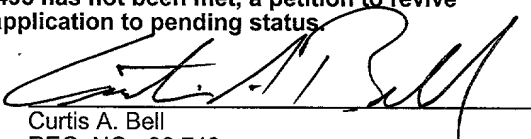
\$130.00

| CLAIMS | NUMBER FILED | NUMBER EXTRA | RATE | | |
|---|--------------|--------------|------------|---------------|--------|
| Total Claims | 20 -20 = | 0 | X \$22.00 | \$0.00 | |
| Independent Claims | 2 - 3 = | 0 | X \$80.00 | \$0.00 | |
| Multiple dependent claims(s) (if applicable) | | 0 | + \$260.00 | \$0.00 | |
| TOTAL OF ABOVE CALCULATIONS = | | | | \$970.00 | |
| Reduction by 1/2 for filing by small entity, if applicable. Verified Small Entity must also be filed. (Note 37 CFR 1.9, 1.27, 1.28). | | | | \$0.00 | |
| SUBTOTAL = | | | | \$970.00 | |
| Processing fee of \$130.00 for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f)). | | | | \$ 0.00 | |
| TOTAL NATIONAL FEE = | | | | \$970.00 | |
| Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). | | | | | |
| \$ 40.00 per property + | | | | \$ | |
| TOTAL FEES ENCLOSED = | | | | \$970.00 | |
| | | | | Amount to be: | |
| | | | | refunded | \$ |
| | | | | charged | \$0.00 |

- a. ☒ A check in the amount of \$970.00 to cover the above fees is enclosed.
- b. ☐ Please charge my Deposit Account No. 08-1650 in the amount of 60.00 to cover the above fees. A duplicate copy of this sheet is enclosed.
- c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 08-1650. A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:
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 206 Sixth Avenue
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 Curtis A. Bell
 REG. NO.: 36,742

001001 21670960

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PATENTS

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants: Vasilios KANELLOPOULOS, Isabelle LOUIS-JOSEPH-DOGUE,
Vincent Daniel McGINNISS, Durvodham MANGARAJ
and Tomoki Tsuchiva NAKAMURA

Serial No.: 09/Not yet assigned

Filed: On even date herewith

For: A POLYMERIC COMPOSITION FOR FRICTION

PRELIMINARY AMENDMENT

Assistant Commissioner for Patents
Washington, D.C. 20231

Dear Sir:

Prior to the calculation of fees and the examination of the above-identified application,
kindly amend the application as follows:

AMENDMENT

In the Claims:

Kindly amend the following claims:

5. (Amended) [A] The polymeric composition according to [any of the preceding
claims, in which] claim 1, wherein the organopolysiloxane resin (II) containing terminal silanol
groups is a hydroxy phenyl alkyl silicone resin.

14. (Amended) [The use of] A method of constructing brake pads comprising the step
of utilizing the polymeric composition [of any of claims 1 to 6] according to claim 1 as a
substrate [for brake pads].

Kindly add new claims 15-20 as follows:

15. (New) The polymeric composition according to claim 2, wherein the
organopolysiloxane resin (II) containing terminal silanol groups is a hydroxy phenyl alkyl
silicone resin.

EXPRESS MAIL CERTIFICATE
No. EL46510381345

I hereby certify that this paper or fee is being
deposited with the U.S. Postal Service using
"Express Mail-Post Office to Addressee"
service under 37 CFR 1.10 and addressed to the
Commissioner of Patents and Trademarks,
Washington, D.C. 20231 on 08-09-2000

Kulyn Conrad

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16. (New) The polymeric composition according to claim 3, wherein the organopolysiloxane resin (II) containing terminal silanol groups is a hydroxy phenyl alkyl silicone resin.

17. (New) The polymeric composition according to claim 4, wherein the organopolysiloxane resin (II) containing terminal silanol groups is a hydroxy phenyl alkyl silicone resin.

18. (New) A method of constructing brake pads comprising the step of utilizing the polymeric composition according to claim 2 as a substrate.

19. (New) A method of constructing brake pads comprising the step of utilizing the polymeric composition according to claim 3 as a substrate.

20. (New) A method of constructing brake pads comprising the step of utilizing the polymeric composition according to claim 4 as a substrate.

In the Specification:

Kindly amend the specification as follows:

On page 1, before line 1 , insert:

--- Title of the Invention ---.

On page 1, after line 1, insert:

--- Cross-Reference to Related Applications

Not Applicable.

Statement Regarding Federally
Sponsored Research or Development

Not Applicable.

Background of the Invention

1. Field of the Invention ---.

On page 1, between lines 11 and 12, kindly insert:

--- 2. Description of the Prior Art ---.

On page 1, between lines 23 and 24, kindly insert:

--- Brief Summary of the Invention ---.

On page 2, between lines 16 and 17, kindly insert:

--- Detailed Description of the Invention ---.

On page 10, line 1, kindly delete "CLAIMS", and substitute therefor:

--- Claims

What is Claimed is: ---.

After page 12, kindly insert the following for the Abstract, also submitted as a separate page:

---Abstract of the Disclosure

The polymeric composition for friction elements comprises a co-polymer between (I) a resin containing phenolic groups and a reticulation agent, and (II) an organopolysiloxane resin containing terminal silanol groups. A part at least of the phenolic groups is bound to the terminal silanol groups. A process of the preparation of the above polymeric composition may comprise the following steps: a) mixing (I) a resin containing the phenolic groups and the reticulation agent, (II) resin containing the terminal silanol groups, and (III) an epoxy resin or the epoxidised organopolysiloxane; b) curing the mixture for a period of time sufficient to complete substantially the reaction between the phenolic groups and the terminal silanol groups, c) post-heating the product obtained under b).---

REMARKS

Claims 1-20 are pending in the above-identified application.

Claims 4 and 15 have been amended to more particularly point out and distinctly claim the subject matter which Applicants regard as the invention and to eliminate multiple dependency and the associated fee therewith.

Claims 15-20 have been added to more particularly point out and distinctly claim the subject matter which Applicants regard as the invention.

An Abstract of the Disclosure has been added and is submitted herewith on a separate page.

CONCLUSION

Favorable action is most earnestly solicited.

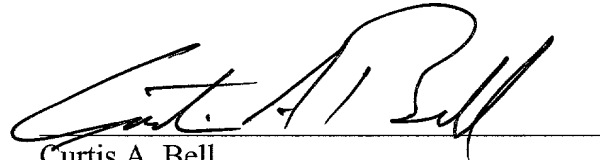
If the Examiner has any questions, or wishes to discuss this matter, please contact the undersigned at the telecommunication numbers listed below.

Respectfully submitted,

Vasilios KANELLOPOULOS,
Isabelle LOUIS-JOSEH-DOGUE,
Vincent Daniel McGINNISS,
Durvodham MANGARAJ
and Tomoki Tsuchiva NAKAMURA

Date: AUGUST 9, 2000

By:



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001021-2889589

-Abstract of the Disclosure

The polymeric composition for friction elements comprises a co-polymer between (I) a resin containing phenolic groups and a reticulation agent, and (II) an organopolysiloxane resin containing terminal silanol groups. A part at least of the phenolic groups is bound to the terminal silanol groups. A process of the preparation of the above polymeric composition may comprise the following steps: a) mixing (I) a resin containing the phenolic groups and the reticulation agent, (II) resin containing the terminal silanol groups, and (III) an epoxy resin or the epoxidised organopolysiloxane; b) curing the mixture for a period of time sufficient to complete substantially the reaction between the phenolic groups and the terminal silanol groups, c) post-heating the product obtained under b).

A POLYMERIC COMPOSITION FOR FRICTION ELEMENTS

The invention is concerned with a polymeric composition for friction elements having remarkable properties with regard to temperature and contact with water. Although the invention will be described in more details with relation to brake pad or brake linings, it should be understood that it may be used in any application in which friction properties have to remain stable with increasing temperature and with water, such brakes and clutches for vehicles and machine tools. The brake pad is one example in which heat and water are of a prime importance due to a possible overheating if the braking action is applied for an extended period of time, during which moreover water may come in contact with the pads.

Preparations or compositions for friction elements for use in brake pads and other applications are known. One example is a mixture in which a phenolic resin and an organopolysiloxane or silicon resin are mixed with a crosslinking agent and described for instance in EP-0 456 490 and JP-63-251 452.

However, according to IR analysis, this mixture appears to be basically a simple mixture of the original phenolic resin and the product of the homoreaction between the silicon resin and itself. This means in particular that the reaction involved do not lead to specific interactions of the phenolic hydroxy groups with the silicon, most of the phenolic groups remaining as such, i.e. as free phenolic groups. Hydrophilic properties are therefore retained together with a relatively high capacity of water absorption, which in turn is affecting strongly the friction characteristics of the product.

The object of the invention is therefore to make the reaction between a phenolic resin and an organopolysiloxane or silicon resin follow a different way, resulting in a actual co-reaction or condensation by co-polymerisation between the phenolic groups and the silanols groups of the silicon in Si-O-C and C-O-C bonds. A part at least of the free phenolic groups of the starting phenolic are consumed in such bonds and will not longer be available for water absorption. The reaction product will lose its hydrophilic properties and the water which may come in contact with said product will not be absorbed, yielding a composition with improved friction properties even under wet conditions.

- 2 -

Another object of the invention is to prepare a composition with superior heat resistance.

Another object of the invention is to prepare a composition with improved wet conditions performance.

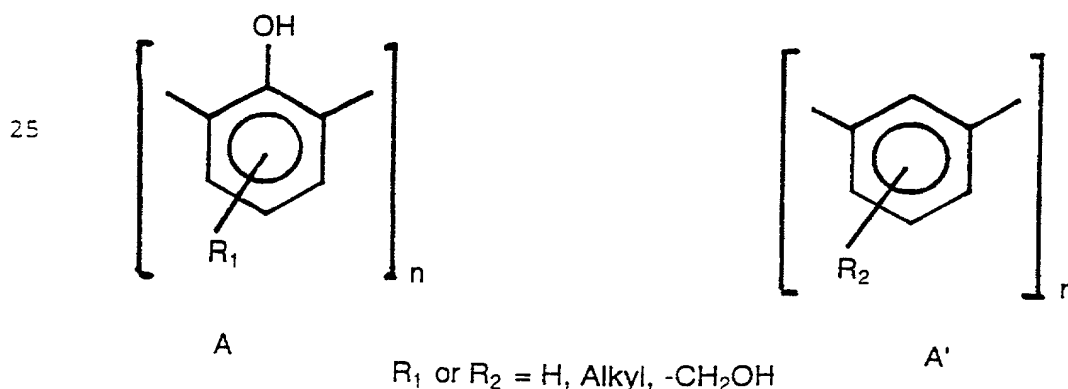
5 In other words, the invention relates to a polymeric composition for friction elements, comprising a co-polymer between (I) a resin containing phenolic groups and a reticulation agent and (II) an organopolysiloxane resin or silicon containing terminal silanol groups, a part of the phenolic groups being bound to the terminal silanol groups.

10 Preferably, the resin containing phenolic groups is from 50 to 80 % and the organopolysiloxane resin containing terminal silanol groups is from 8 to 25 % by weight of the total starting mixture.

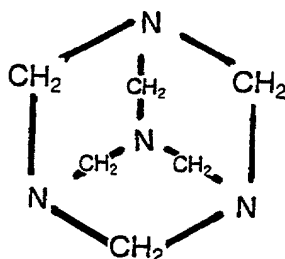
The starting resin comprising phenolic groups may also comprise terminal non aromatic alcoholic groups, a part at least of the terminal non aromatic alcoholic groups being also bound to the terminal silanol groups.

The reticulation agent may be an amine, such as an hexamine.

In one embodiment of the invention, the reticulation agent is an hexamine and is already present as a mix in a resin containing phenolic groups. Such a starting material is for instance that sold under the name of [®]Xylox by Mitsui
20 Toatsu Chemicals. In this commercial product, the resin containing phenolic groups is of the general formula (A) and may include moieties of a general formula (A'), and contains hexamine (B) in a proportion between 8 and 12 % by weight.



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5

Other starting materials of the same sort may be used as well, such as[®]Novalak type of resins

10 The other compound, namely an organopolysiloxane resin containing terminal silanol groups may be an hydroxy phenyl alkyl silicone resin or methyphenylsiloxane for instance.

The invention relates as well to a process for the preparation of the polymeric composition, comprising the following steps :

15 a) mixing (I) a resin containing the phenolic groups and the reticulation agent, (II) a resin containing the terminal silanol groups, and (III) an epoxy resin or the epoxidised organopolysiloxane

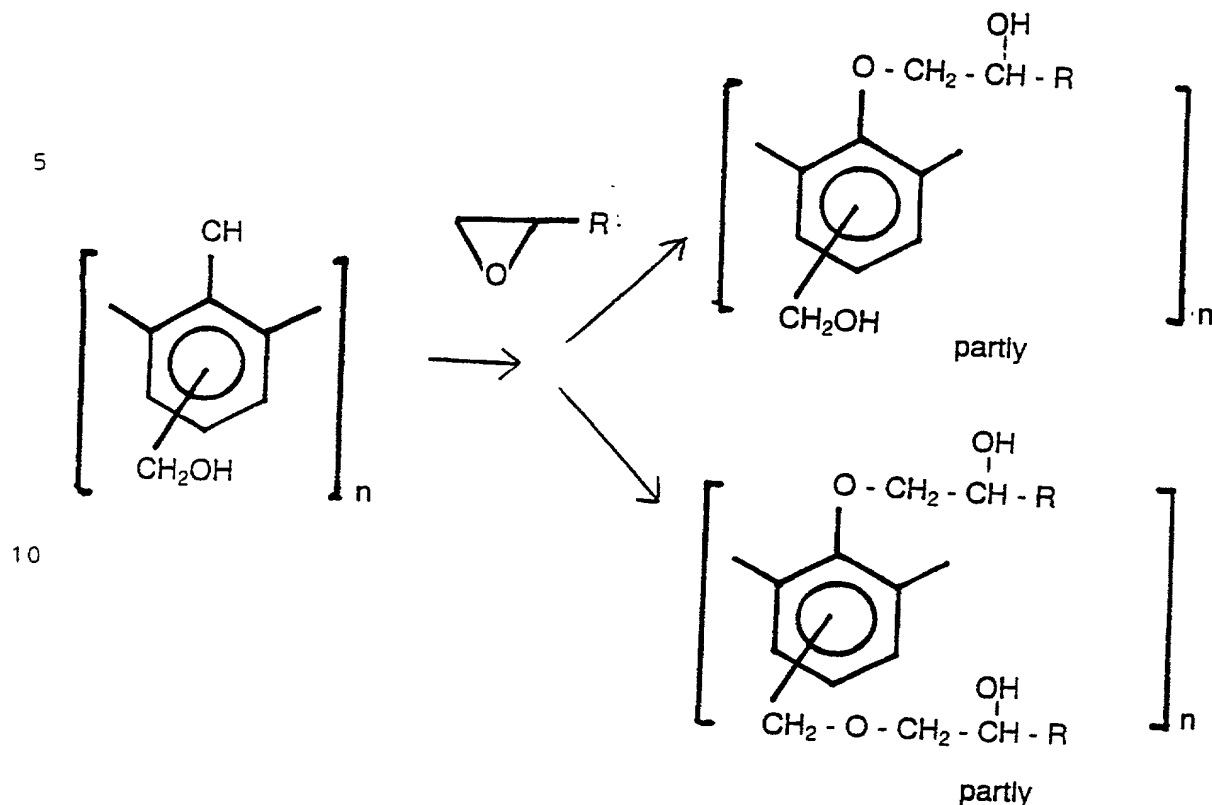
b) curing the mixture for a period of time sufficient to substantially complete the reaction between the phenolic groups and the terminal silanol groups,

c) post-heating the product obtained under b).

20 It should be noted that the reaction is made in the presence of an epoxy resin or an epoxidised organopolysiloxane. This will push the reaction towards the way of a condensation or co-reaction leading to a copolymer rather than a simple homoreaction between the silicone resin and itself as mentioned above for the prior art.

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Such a reaction involving the epoxy resin may be symbolised as follows :



15 The silicone resin is present in the starting mixture from 10 to 20 % by weight , preferably around 20%. The epoxy resin may be for instance of [®]Ciba-Geigy (GT 7071) type and may be present in the starting mixture from 20 to 40 % by weight. The epoxidised organopolysiloxane may be for instance a polydialkylsiloxane and may be present in the starting mixture from 3 to 10%, but

20 preferably around 5%

To make easier the blending of the starting resins, said resins are preferably in a form of powder with a particle size distribution of not more than 400 μm , preferably below 300 μm for a compound such as [®]Xylok cited above, and 200 μm for silicone.

25 The mixing step a) which may be held as well as a step for forming or shaping the end product is preferably conducted in a mould at a temperature not exceeding 50°C.

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In general, the curing step b) is conducted under a pressure of at least 50 atm and a temperature from 80 to 160°C and may be divided in a number of cycles permitting the degassing of the reaction mixture. In this case each degassing cycle is most preferably performed in sequence at increasing pressures and temperatures.

As to the post-heating step c) the temperature is advantageously of at least 200°C, under atmospheric pressure.

The various objects and advantages of the invention will become apparent with regard to the following non limitative examples.

EXAMPLES 1 TO 7

From a starting blend containing 20% epoxy resin GT 7071, 10% silicone resin 6-2230 and 70%[®]Xylok, samples of 10 x 60 mm (table 1) were fabricated following the conditions described below.

- Step 0: At least 60 s at the curing temperatures without pressure
- Step 1: 5 degassing cycles with a pressure of 146 atm (6 s on, 10 s off) at the curing temperatures for 5, 12, 17.5, 23, or 30 minutes respectively at the curing temperature with a pressure of 183 atm.
- Step 2: 10 minutes at 160°C with 3 degassing cycles (6 s on, 10 s off)

Table 1

| Example | Curing temperature (°C) | Time (min) |
|---------|-------------------------|------------|
| 1 | 80 | 23 |
| 2 | 80 | 12 |
| 3 | 150 | 23 |
| 4 | 150 | 12 |
| 5 | 115 | 5 |
| 6 | 115 | 30 |
| 7 | 115 | 17.5 |

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Specimens of these formulations were submitted to different post-curing temperatures of 200°C and 240°C and processed with a brake pad with the usual additional ingredients, to form specific formulations for water tests.

The water absorption was tested using a method, where a 10 µl water drop is deposited at the surface of the sample and the time for absorption is recorded (table 2). On a [®]Teflon surface, which was used as a reference, a 10 µl drop was evaporated in 60 minutes.

Table 2

| Examples | Curing Temperature (°C) | Post-curing at 200°C | Post-curing at 240°C |
|----------|-------------------------|----------------------|----------------------|
| 2 | 80 | 55 min | 61 min |
| 3 | 115 | 62 min | 74 min |
| 6 | 150 | 63 min | 67 min |

The results showed that the time needed for the water to disappear corresponds to almost that for its evaporation, which confirms that the water absorption for these resin formulations is very low.

After a heat treatment to 350°C, which was intended to simulate heating and over heating by breaking, the following comparative results were obtained for formulations post-cured at 240°C.

With specific formulations (resin and other conventional brake pad ingredients) prepared for water tests, the time needed for water to disappear (absorber or evaporated) for pure [®]Xylok correspond to 12 s and to 10s after heating at 350°C for 1h and 2 h respectively. The corresponding times for the specific formulations according to the invention, were at 407 s after 1h heating at 350°C and 186 s after 2h heating at 350°C.

Several reactions could be expected between the different compounds of the formulations:

- the OH end groups of the Xylok could react with the epoxy groups of GT 7071 resin allowing the formation of a C-O-CH₂-CHOH- bond,

- the OH end groups of the Xylok could react with those of the polydimethyl siloxane 6-2230 leading to the formation of a Si-C-O bond.

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Infra-red evidence of the formation of those groups should be the reduction of the phenyl-CH₂-OH characteristic band near 1010 cm⁻¹ as well as the appearance of the typical bands of Si-C-O bond near 1100 cm⁻¹ (asymmetric stretching vibrations) and C-O-CH₂ bond near 1040 cm⁻¹.

- 5 The time and temperature of exposure before curing (step 1) are important to the extent of reaction between silicone-hydroxyl and [®]Xylok-hydroxyl groups. Hence, the change in the peaks were studied as a function of pre-curing temperature and time. Table 3 (reference bands with regards to starting resins) and Table 4 below summarise the characteristic bands for each IR spectrum.

10

Table 3

| Bands/ Samples | Epoxy groups 835 cm ⁻¹ | Si-O-C 850 cm ⁻¹ | Si-OH 900 cm ⁻¹ | ϕ CH ₂ OH 1010 cm ⁻¹ | Si-O-C or C-O-C- 1100 cm ⁻¹ | -C = O 1650 cm ⁻¹ | -CH- 3000 cm ⁻¹ | -OH 3100-3600 cm ⁻¹ |
|--------------------|---|-----------------------------------|----------------------------------|---|--|------------------------------------|----------------------------------|--------------------------------------|
| [®] Xylok | none | none | none | strong | weak | none | strong | strong |
| Silicone resin | none | none | strong | none | strong | none | weak | weak |
| Epoxy resin | strong | none | none | none | weak | none | strong | weak |

15

Table 4 relates to formulations where step 0 was conducted at 80°C, with comparison to samples where step 0 was conducted of 12 min at 80°C , respectively with no curing or post-curing (3rd column in table).

Table 4

| Conditions | | | Epoxy groups | Si-O-C | Si-OH | ϕ CH ₂ OH | Si-O-C or C-O-C | -C = O | -CH- | -OH |
|------------------|--------|-----------------|-----------------|-------------|-------------|------------------------------|-----------------------|--------------|--------------|-----------------------|
| Reaction time | Curing | Post- curing | 835 cm-1 | 850 cm-1 | 900 cm-1 | 1010 cm-1 | 1100 cm-1 | 1650 cm-1 | 3000 cm-1 | 3100- 3600 cm-1 |
| 12 min | no | no | strong | none | weak | strong | strong | none | strong | strong |
| 12 min | no | no | stronger | none | weak | smaller | strong | none | stronger | stronger |
| 12 min | 165°C | no | stronger | none | smaller | smaller | stronger | none | strong | lower |
| 12 min | 165°C | 240°C | stronger | none | smaller | smaller | stronger | strong | strong | strong |
| 23 min | 165°C | 240°C | stronger | none | smaller | smaller | stronger | stronger | strong | strong |

EXAMPLES 8 TO 14

From a starting blend containing 5% of epoxidised solution of Dow Corning sold under the name of Additive 23, 20% silicone resin 6-2230 and 75%[®]Xylok, 10 x 60 mm samples (table 1) were fabricated following the conditions described below.

Step 0: At least 1.5 min at the curing temperatures without pressure

Step 1: 5 degassing cycles with a pressure of 148 atm (6 s on, 10 s off) at the curing temperatures. 12, 17.5 or 30 minutes at the curing temperature with a pressure of 183 atm.

Step 2: 10 minutes at 160°C with 3 degassing cycles (6 s on, 10 s off)

Table 5

| Examples | Curing temperature (°C) | Time (min) |
|----------|-------------------------|------------|
| 8 | 140 | 23 |
| 9 | 140 | 12 |
| 10 | 160 | 23 |
| 11 | 160 | 12 |
| 12 | 150 | 5 |
| 13 | 150 | 30 |
| 14 | 150 | 17.5 |

Specimens of these formulations were submitted to different post-curing temperatures of 200°C and 240°C and processed with a brake pad with the usual additional ingredients, to form specific formulations for water tests.

The water absorption was again tested as for the formulations of the previous examples 1 to 7 using the above method of water droplet. As a result., the formulations as presented below, showed similar behaviour to that of the previous examples.

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Table 6

| Examples | Curing Temperature (°C) | Reaction time (min) | Post-curing at 200°C | Post-curing at 240°C |
|----------|-------------------------|---------------------|----------------------|----------------------|
| 9 | 140 | 12 | 70 min | 72 min |
| 10 | 150 | 17.5 | 80 min | 77 min |
| 14 | 160 | 23 | 77 min | 66 min |

The results showed that the time needed for the water to disappear corresponds to almost that for its evaporation, which confirms that the water absorption for these resin formulations is very low.

After a heat treatment to 350°C, which was intended to simulate heating and over heating by breaking, the following comparative results were obtained for formulations post-cured at 240°C.

With specific formulations (resin and other conventional brake pad ingredients) prepared for water tests, the time needed for water to disappear (absorbed or evaporated) for pure ©Xylox correspond to 12 s and to 10 s after heating at 350°C for 1h and 2 h respectively. The corresponding times for the specific formulations according to the invention, were 1'972 s after 1 h heating at 350°C and 1'832 s after 2 h heating at 350°C.

Also, if time needed for the water to disappear (absorbed or evaporated) correspond to 100% for the respective above inventive formulations as crude samples (no treatment to 350°C), then after 1 h of heating to 350°C these specific formulations for water tests were at 75 %, 98 % and 98 % respectively for pure ©Xylox, Mitsui product (©Xylox plus Si) and the inventive formulations.

However, after 2 h of heating at 350°C, the results were 63 %, 53 % and 87 % respectively for pure ©Xylox, Mitsui product (©Xylox plus Si) and the inventive formulations.

These results indicate the excellent performance and good resistance of the inventive resin formulations to prolonged heat treatments as compared to pure ©Xylox or even the Mitsui product (©Xylox plus Si).

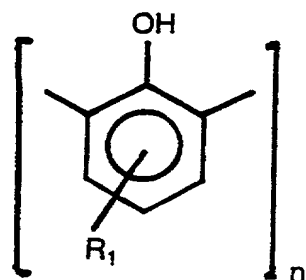
-10-

CLAIMS

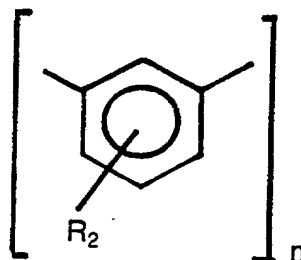
1. A polymeric composition for friction elements which comprises a co-polymer between (I) a resin containing phenolic groups and a reticulation agent (II) an organopolysiloxane resin containing terminal silanol group, a part at least of the phenolic groups being bound to the terminal silanol groups, and an epoxy resin or an epoxidised organopolysiloxane (III).

2. A polymeric composition according to claim 1, wherein the resin comprising phenolic group comprises also terminal non aromatic alcoholic groups, a part at least of the terminal non aromatic alcoholic groups being bound to the terminal silanol groups.

3. A polymeric composition according to any of the preceding claims, in which the resin (I) containing phenolic groups is of general formula (A) and may include moieties of the general formula (A') :



A



A'

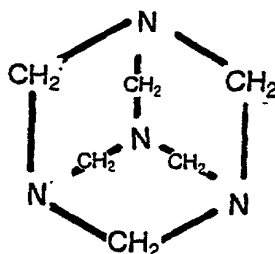
R_1 or $R_2 = \text{H, Alkyl, } -\text{CH}_2\text{OH}$

4. A polymeric composition according to claim 3, in which the resin (I) containing phenolic groups is of general formula (A) and includes moieties of the general formula (A').

5. A polymeric composition according to any of the preceding claims, in which the organopolysiloxane resin (II) containing terminal silanol groups is a hydroxy phenyl alkyl silicone resin.

- 11 -

6. A polymeric composition according to any of the preceding claims, in which the reticulation agent is an hexamine of general formula (B) :



7. A process of preparation of a polymeric composition according to any of the preceding claims, comprising the following steps :

a) mixing (I) a resin containing the phenolic groups and the reticulation agent, (II) resin containing the terminal silanol groups, and (III) an epoxy resin or the epoxidised organopolysiloxane

b) curing the mixture for a period of time sufficient to complete substantially the reaction between the phenolic groups and the terminal silanol groups,

c) post-heating the product obtained under b).

8. A process according to claim 7. in which the mixing step a) is conducted at a temperature not exceeding 50°C.

9. A process according to claim 8, in which the curing step b) is conducted under a pressure of at least 50

atm and the temperature is from 80 to 160°C.

10. A process according to claim 7. in which the curing step b) is divided in a number of cycles permitting the degassing of the reaction mixture.

11 A process according to claim 10, in which each degassing cycle is conducted in sequence at increasing pressure and temperature.

12 A process according to claim 7 in which the post-heating step c) is conducted at a temperature of at least 200°C under atmospheric pressure.

-12-

13. A process according to claim 7 in which the starting resins are in a form of powder with a particle size distribution of not more than 400 μm
14. The use of the polymeric composition of any of claims 1 to 6 as a substrate for brake pads

[illegible]

COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY

Docket No. 6-1034-040

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and was amended on August 9, 2000 (if applicable).

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and was amended under PCT Article 19 on _____ (if applicable).

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Priority Claimed

| (Number) | (Country) | (Day/Month/Year Filed) | Yes No |
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| <u>EP 98420024.6</u> | <u>EP</u> | <u>09 February 1998</u> | <u>X</u> |

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Full name of sole or first inventor Vasilios KANELLOPOULOS
Inventor's signature [Signature]
Date 18 September 2000
Residence CH-1256 Troinex, SWITZERLAND
Citizenship Switzerland
Post Office Address 6, chemin Sous-le-Cret
CH-1256 Troinex, SWITZERLAND

Full name of second inventor Isabelle LOUIS-JOSEPH-DOGUE
Inventor's signature [Signature]
Date _____
Residence F-74100 Annemasse, FRANCE
Citizenship France
Post Office Address 43, rue du Dr Coquand
F-74100 Annemasse, FRANCE

Full name of third inventor _____ Vincent Daniel McGINNISS _____
Inventor's signature ✓ _____
Date ✓ _____
Residence _____ Sunbury, Ohio 43074 U.S.A. _____
Citizenship _____ U.S.A. _____
Post Office Address _____ P.O. Box 702 _____
_____ Sunbury, Ohio 43074 U.S.A. _____

Full name of fourth inventor _____ Duryodhan MANGARAJ _____
Inventor's signature ✓ _____
Date ✓ _____
Residence _____ Dublin, Ohio 43017 U.S.A. _____
Citizenship _____ U.S.A. _____
Post Office Address _____ 7828, Blackjack Court _____
_____ Dublin, Ohio 43017 U.S.A. _____

Full name of fifth inventor _____ Tomoki Tsuchiya NAKAMURA _____
Inventor's signature ✓ _____
Date ✓ _____
Residence _____ Narita, Chiba 892-11, JAPAN _____
Citizenship _____ Japan _____
Post Office Address _____ Narita, Chiba 892-11, JAPAN _____

COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY

Docket No. 6-1034-040

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| EP 98420024.6 | EP | 09 February 1998 | X |
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Inventor's signature _____

Date _____

Residence CH-1256 Troinex, SWITZERLAND

Citizenship Switzerland

Post Office Address 6, chemin Sous-le-Cret
CH-1256 Troinex, SWITZERLAND

Full name of second inventor Isabelle LOUIS-JOSEPH-DOGUE

Inventor's signature [Signature]

Date 28/08/2000

Residence F-74100 Annemasse, FRANCE

Citizenship France

Post Office Address 43, rue du Dr Coquand
F-74100 Annemasse, FRANCE PRX

09601912-120100

Full name of third inventor Vincent Daniel McGINNISS
Inventor's signature _____
Date _____
Residence Sunbury, Ohio 43074 U.S.A.
Citizenship U.S.A.
Post Office Address P.O. Box 702
Sunbury, Ohio 43074 U.S.A.

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Inventor's signature _____
Date _____
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Dubin, Ohio 43017 U.S.A.

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Inventor's signature _____
Date _____
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Citizenship Japan
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COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY

Docket No. 6-1034-040

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Inventor's signature ✓

Date ✓

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Inventor's signature ✓

Date ✓

Residence F-74100 Annemasse, FRANCECitizenship FrancePost Office Address 43, rue du Dr CoquandF-74100 Annemasse, FRANCE

09601912-120100

3-00
Full name of third inventor Vincent Daniel McGINNISS
Inventor's signature *Vincent Daniel McGinniss*
Date 18 September 2000
Residence Sunbury, Ohio 43074 U.S.A.
Citizenship U.S.A.
Post Office Address P.O. Box 702
Sunbury, Ohio 43074 U.S.A.

Full name of fourth inventor Duryodhan MANGARAJ
Inventor's signature *Duryodhan Mangaraj*
Date ✓
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Citizenship U.S.A.
Post Office Address 7828 Backjack Court
Dubin, Ohio 43017 U.S.A.

Full name of fifth inventor Tomoki Tsuchiya NAKAMURA
Inventor's signature *Tomoki Tsuchiya Nakamura*
Date ✓
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Citizenship Japan
Post Office Address Narita, Chiba 892-11, JAPAN

COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY

Docket No. 6-1034-040

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09601912 "120100

Full name of third inventor Vincent Daniel McGINNISS
Inventor's signature ✓
Date ✓
Residence Sunbury, Ohio 43074 U.S.A.
Citizenship U.S.A.
Post Office Address P.O. Box 702
Sunbury, Ohio 43074 U.S.A.

Full name of fourth inventor 4-W Duryodhan MANGARAJ
Inventor's signature ✓ D. Mangaraj
Date ✓ 9.18.02
Residence Dubin, Ohio 43017 U.S.A.
Citizenship U.S.A.
Post Office Address 7828, Backjack Court
Dubin, Ohio 43017 U.S.A.

Full name of fifth inventor Tomoki Tsuchiya NAKAMURA
Inventor's signature ✓
Date ✓
Residence Narita, Chiba 892-11, JAPAN
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Full name of fifth inventor Tomoki Tsuchiya NAKAMURA
Inventor's signature Tomoki Nakamura
Date ☒ Nov 24, 2000
Residence Narita, Chiba 892-11, JAPAN
Citizenship Japan
Post Office Address Narita, Chiba 892-11, JAPAN JAX